

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/322632523>

Synthesis Features of Iron Oxide Nanopowders with High Magnetic and Sorption Properties

Article in *Materials Science Forum* · January 2018

DOI: 10.4028/www.scientific.net/MSF.915.116

CITATIONS

0

READS

41

5 authors, including:



A. G. Mamalis

National Center for Scientific Research Demo...

626 PUBLICATIONS 4,539 CITATIONS

SEE PROFILE



Д.С. Софронов

Institute for Single Crystals

69 PUBLICATIONS 142 CITATIONS

SEE PROFILE



Vadym Starikov

Kharkiv Polytechnical Institute

56 PUBLICATIONS 155 CITATIONS

SEE PROFILE

Some of the authors of this publication are also working on these related projects:



Mechanical Engineering Research [View project](#)



Halide crystals growth by Skull technique [View project](#)

All content following this page was uploaded by [A. G. Mamalis](#) on 21 January 2018.

The user has requested enhancement of the downloaded file.

Synthesis Features of Iron Oxide Nanopowders with High Magnetic and Sorption Properties

Sergiy LAVRYNENKO^{1,a}, Athanasios G. MAMALIS^{2,b}, Dmitry SOFRONOV^{3,c},
Alexandra ODNOVOLOVA^{3,d} and Vadym STARIKOV^{1,e}

¹National Technical University "Kharkov Polytechnic Institute", Kharkov, Ukraine

²Project Center for Nanotechnology and Advanced Engineering, NCSR "Demokritos",
Athens, Greece

³State Scientific Institution "Institute for Single Crystals", National Academy of Sciences
of Ukraine, Kharkov, Ukraine

^alavr@kpi.kharkov.ua, ^ba.mamalis@inn.demokritos.gr, ^csofronov@isc.kharkov.com,

^dodnovolova.alexandra@gmail.com, ^estarik@kpi.kharkov.ua

Keywords: iron oxide nanoparticles, magnetization, sorption, synthesis

Abstract. The magnetic particles of iron oxides are promising materials for the purification of water from ions of heavy metals and radionuclides. Their advantage compared to other sorbents is the ability to extract by applied magnetic field, which greatly simplifies the task of extraction, separation and processing in cleaning technologies. The aim of this work is investigation of temperature and concentration of iron in the solution effect on the phase composition, nanoparticle size and their magnetization.

Phase magnetite in the sample increases with increasing temperature and the magnetization decreases slightly with increasing the initial concentration of iron in solution. We found that regardless of the conditions of deposition formed spherical particles whose average size ranges from 7 to 15 nm. The sorptive capacity of the particles is virtually independent of the phase composition and for cobalt is about 18 mg/g. For sorption-based material magnetic particles Fe₃O₄ recommended to carry out the deposition process at a temperature not lower than 80°C. The concentration of iron in solution must be within 0,15–0,3M. The particles obtained contain in their composition at least 90 wt.% of magnetite phase and are characterized by a magnetization in the range of 65–70 A·m²/kg. Also in the paper is comparing efficiency of extraction and sorption capacity for cobalt particles by different phase of magnetite and hematite.

Introduction

Sorption extraction is one of the most effective and simple methods of purification of natural and waste waters from heavy metals and radionuclides [1, 2]. Polymers [3], natural materials, oxides [4] were proposed to use as sorbents. In the latter group of materials iron oxides should be noted as components of effective sorption materials. Their advantage over other compounds consists in low cost of precursors, simplicity of synthesis, and in the fact that they exert no toxic effects on the human body [5].

Typically, the particles of iron oxide are produced by deposition from aqueous solution with a subsequent heat treatment at 200–600°C. [6, 7].

It is well known that the functional properties of materials are largely determined by particle size and shape. For example, increasing the specific surface area of the sorbent leads to an increase in its sorptive capacity. Thus, in case of α -Fe₂O₃ of a specific surface of 130 m² g⁻¹ the sorption capacity (at pH 3) for arsenic(V) and chromium(VI) was 51 and 30 mg g⁻¹, respectively. A decrease in the specific surface area of up to 2 m² g⁻¹ led to a decrease in the sorption capacity of up to 0.30 and 0.37 mg g⁻¹, respectively [8].

Thus, an issue of the effect of the conditions of particles producing on their morphological and functional characteristics is a topical. The nature of the anions is one of the factors exerting a significant effect on the formation of particles in deposition from aqueous solutions [9-12].

Experimental Procedure

Synthesis of Fe₂O₃ particles

Synthesis of Fe₂O₃ particles as follows: 100 ml of a 0.2M precursor salts of iron (III) an aqueous solution of ammonia or ammonium bicarbonate to a predetermined pH ranging from 7 to 12 and stirred on a magnetic stirrer for 30 min. The resulting precipitate was filtered, washed several times with distilled water and dried at room temperature for 24 h, then calcined at 450°C for 1 hour.

Synthesis of Fe₃O₄ particles

Synthesis of Fe₃O₄ particles was performed by precipitation from aqueous solution: 5.99 g FeSO₄•7H₂O and 10.79 g of FeCl₃•6H₂O was dissolved in 40 ml of distilled water. The resulting solution was heated to 60°C and 10 ml of 25% aqueous ammonia solution under constant magnetic stirring. The resulting precipitate was filtered and washed with distilled water. Then dried in air for 24 hours.

The sorption properties

Sorption is carried out in monocomponent heavy metal solution at pH 6.5 with concentration of 10–250 mg/l. In a 100 ml beaker the 50 ml of model solution was adjusted by addition of nitric acid or aqueous ammonia solution to a predetermined pH 6.5. Solution pH was measured by the "150 pH" pH-meter with glass electrode ESL-63-07 and the auxiliary silver chloride electrode EVL1MZ. Then was added 0.1 g of the adsorbent and kept under stirring for 40 minutes. The process of sorption was performed every 10 minutes with pH control in the case of deviations exceeding 0.1 pH units, respectively adjusted to predetermined values. The sorbent was filtered after sorption and the content of metals in the filtrate was determined.

Results and Discussion

X-ray analysis of the sample Fe₂O₃ showed that after annealing at 450°C formed α -Fe₂O₃ (hematite). Unlike samples Fe₂O₃, for Fe₃O₄ samples X-ray data is multiphase and contain magnetite (Fe₃O₄) – 70%, maghemite (γ -Fe₂O₃) – 25% and goethite (FeOOH) – 5%. The presence of impurity phases goethite and maghemite Fe₃O₄ samples associated with aging and is characteristic of the powders obtained from aqueous solutions.

Fig. 1 shows the photomicrographs of particles (a) Fe₂O₃ and (b) Fe₃O₄, obtained from aqueous solution and annealed at 450°C.

It is established that from nitrate solutions formed shapeless large agglomerates of 50 – 200 microns (Fig. 1a), consisting of spherical particles of Fe₂O₃ (specific surface S_{sp} = 150 m²/g). Introduction of chloride ions leads to the formation of solid particles of cubic and oval size of about 0.12 – 0.20 microns (S_{sp} = 30 m²/g). Sodium ion has no significant effect on the formation of particles Fe₂O₃.

Unlike Fe₂O₃, Fe₃O₄ is not observed in the formation of large agglomerates and the powder is composed of spherical with a size of from 0.5 μ m to several microns. The specific surface area of Fe₃O₄ was 130 m²/g.

Fig. 2 shows the degree of extraction of metals from the pH of the particles Fe₂O₃ (Fig. 2(a) and (c)) and Fe₃O₄ (Fig. 2(b) and (d)).

As can be seen from Fig. 2, iron oxide particles effectively recovered europium (97%), Ce (97%) and copper (92%). However, in an acidic medium using Fe₂O₃ particles as sorbents can achieve high degrees of recovery of these metals as compared with Fe₃O₄ particles.

The maximum extraction for Fe₂O₃ is observed at pH 5.5, and for Fe₃O₄ is optimal pH 6.5. Efficiency of extraction of cobalt and strontium increases with increasing pH and reached maximum values at pH 9.

The highest efficiency of extraction occurs on the particles of Fe₃O₄ and 95% to 68% cobalt, and strontium.

Furthermore, for strontium in the pH range 4 – 7, depending observed additional peak recoveries in the range of pH 5.2 – 5.7, which is more pronounced on Fe₂O₃ particles, see Fig. 2(a).

The results of calculation by Langmuir's method shown in Table 1. As can be seen from the data, the size of the sorption capacity Fe₂O₃ particles with 150 m²/g specific surface for all investigated metals are higher than the sorbents based on Fe₃O₄ with 130 m²/g specific surface.

Table 1 Parameters of sorption isotherms of europium, cerium and copper particles Fe₂O₃ and Fe₃O₄

Metal	Fe ₂ O ₃			Fe ₃ O ₄		
	K	A _∞ , [mg/g]	R ² , [%]	K	A _∞ , [mg/g]	R ² , [%]
Eu	0.007	21.3	89.85	0.024	19.7	92.97
Ce	0.346	9.2	94.83	0.162	7.5	90.52
Cu	0.029	15.7	94.12	0.039	11.66	93.57

The highest sorption capacity is observed for the europium – 21.3 mg/g and 19.7 mg/g on the particles of Fe₂O₃ and Fe₃O₄, respectively, and the lowest - cerium on the particles of Fe₂O₃ (9,2 mg/g) and Fe₃O₄ particles (7.5 mg/g).

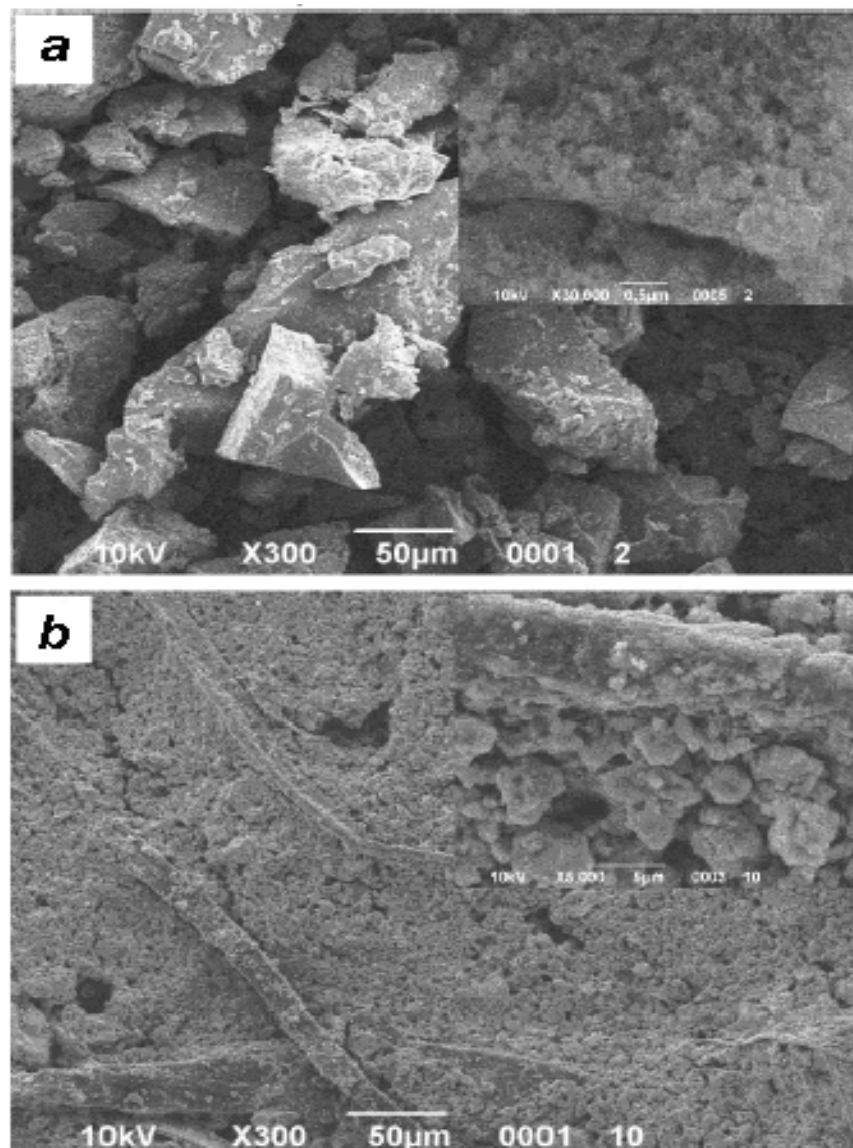


Fig. 1 Micrographs of particles (a) Fe₂O₃, (b) Fe₃O₄ annealed at 450°C

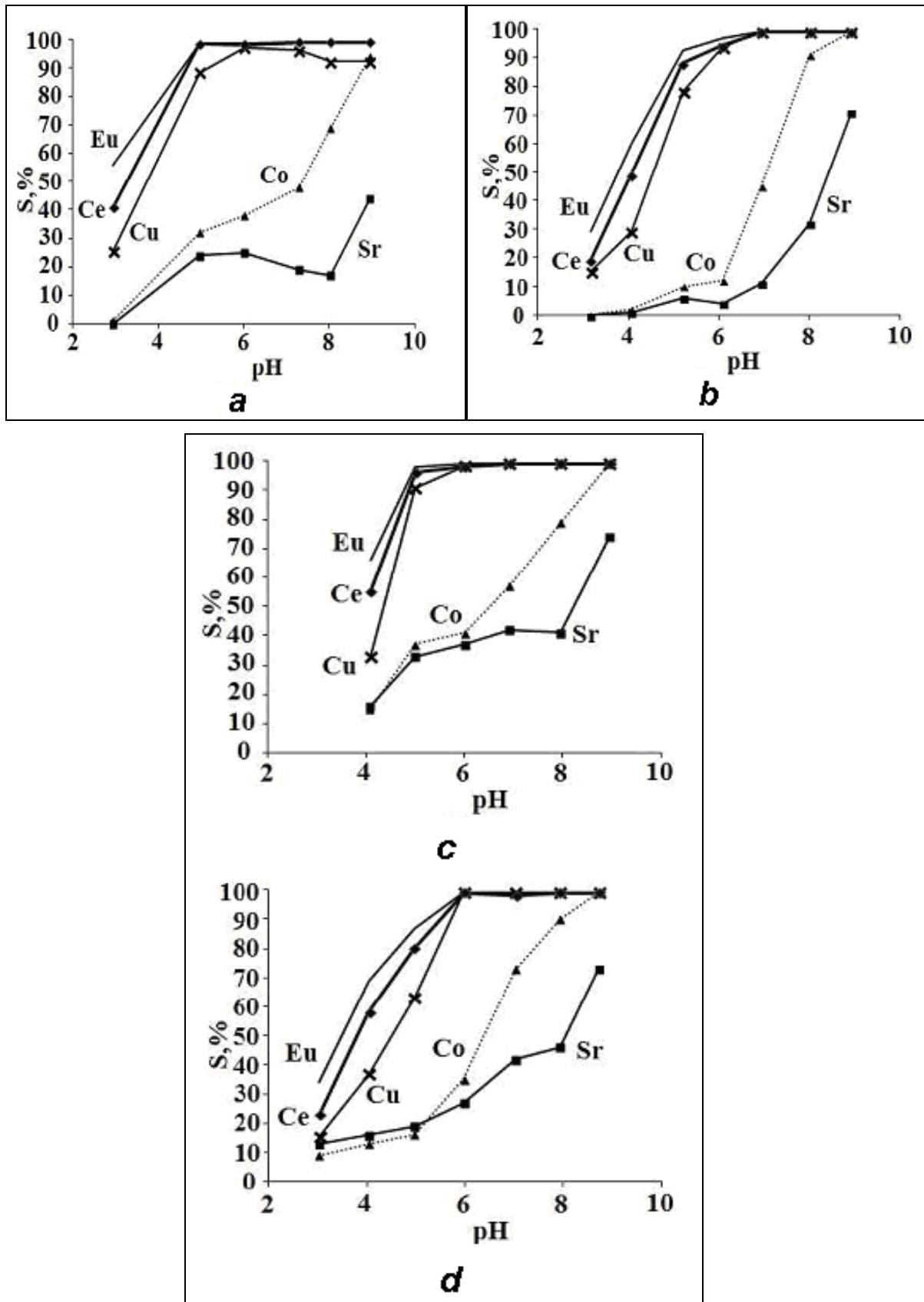


Fig. 2 Dependence of the degree of extraction of metals from the pH of the particles: (a) and (c) Fe_2O_3 ; (b) and (d) Fe_3O_4

Summary

- (a) The effect of the deposition conditions (temperature, concentration of iron in solution) on the phase composition, particle size and magnetization. A comparison of the efficiency of extraction and sorption capacity of heavy metals in phase magnetite and hematite.
- (b) It was found that the formation of magnetite phase is significantly affected by the deposition temperature. The higher the temperature, the greater the amount of magnetite phase in the sample. Carrying out the synthesis at 90°C in a 0.15M solution promotes formation of iron powder containing phase magnetite (Fe₃O₄) 100 wt. %
- (c) Regardless of the deposition conditions form spherical particles having an average size ranging from 7 to 15 nm.
- (d) Raising the deposition temperature and reducing the concentration of iron in the solution contributes to obtaining a powder with larger particles that are characterized by higher value of magnetization.

References

- [1] S.W. Park and C.P. Huang, *J. Colloid a. Interface Sci.*, 128(1)(1989), 245-257
- [2] G.V. Myasoedova and V.A. Nikishina, *Ros. Khim. Zh.*, 1(5)(2006), 55-63
- [3] I.A., Kovalev, N.M. Sorokina and G.I. Tsizin, *Vestn. MGU. Khimiya*, 41(5)(2000), 30-314
- [4] A.S. Shilina and V.K. Milinchuk, *Sorb. Khromatogr. Prots.*, 10(2)(2010), 237-245
- [5] Yao-Hui Huang, Chan-Li, Hsueh, Hui-Pin Cheng, et al., *J. Materials*, 144(2007), 406-411
- [6] Y. Dong and X. Tian X, *J. Non-Crystall. Solids*, 356(2010), 1404-1407
- [7] O.S. Petrova, A.E. Chekanova, E.A. Gudilin, et al., *Int. Sci. J. Alternative Energy and Ecology*, 45(1)(2007), 70-73
- [8] Chang-Yan, Cao, Jin Qu, Wen-Sheng, Yan, et al., *Langmuir*, 28(2012), 4573-4579
- [9] D.S. Sofronov, E.M. Sofronova, V.N. Baumer, et al., *Advanced Powder Technol.*, 24(6)(2013), 1017-1022
- [10] D.S. Sofronov, E.M. Sofronova, V.N. Baumer, et al., *Functional Materials*, 18(2011), 523-528
- [11] D.S. Sofronov, D.S., N.N. Kamneva, A.V. Bulgakova, et al., *J. Biol. Phys. and Chem.*, 13(2013), 85-89
- [12] A.M. Odnovolova, D.S. Sofronov, P.V. Mateichenko, et al. *Russian J. of Appl. Chem.*, 87(8) (2014), 1060-1064